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TECHNICAL REPORT No. 7

to

THE OFFICE OF NAVAL RESEARCH

Contract NONR 609(28)

FINAL SUMMARY REPORT

of

RESEARCH INVESTIGATIONS CONDUCTED UNDER THE PROVISIONS

of

Contract NONR 609(28)

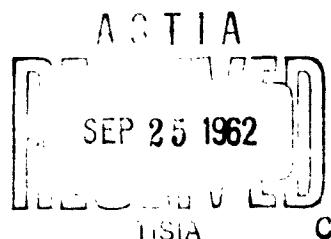
by

W. D. Robertson, Principal Investigator

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Hammond Metallurgical Laboratory
Yale University
New Haven, Conn.

August, 1962



FINAL SUMMARY REPORT OF INVESTIGATIONS

The investigations performed under the terms of Contract NONR 609(28) have continued and extended the work begun under a previous contract, NONR 305(00). Contract NONR 609(28) began July 15, 1958 and terminated July 15, 1961.

All of the investigations have been reported in detail in technical reports to the Office of Naval Research under the following titles and dates:

<u>Technical Report</u>	<u>Title</u>	<u>Date</u>
1	"The Mechanism of Hardening in Aged Aluminum-Copper Alloys", and "Dispersed Particle Hardening of Aluminum-Copper Alloy Single Crystals"	October 1960
2	"A Unified Structural Mechanism for Intergranular and Transgranular Corrosion Cracking"	December 1960
3	"The Mechanism of Hydrogen Embrittlement Observed in Iron-Silicon Single Crystals"	September 1961
4	"Martensitic Transformation in Single Crystals of Iron Alloys"	February 1962
5	"Direct Observation and Analysis of Crack Propagation in Iron-3% Silicon Single Crystals"	February 1962
6	"Martensitic Transformation and Plastic Deformation in Iron Alloy Single Crystals"	July 1962

The substance of each of these reports has been published or submitted for publication as shown in the following bibliography:

1. "The Mechanism of Hardening in Aged Aluminum-Copper Alloys", D. Dew-Hughes and W. D. Robertson - *Acta Met.*, 8, 147, (March 1960)
2. "Dispersed Particle Hardening of Aluminum-Copper Alloy Single Crystals" D. Dew-Hughes and W. D. Robertson, - *Acta Met.*, 8, 156, (March 1960)
3. "A Unified Mechanism of Intergranular and Transgranular Cracking", A. S. Tetelman and W. D. Robertson, American Society for Metals Symposium, Mechanism of Strengthening in Solids, Cleveland (1962)

4. "The Mechanism of Hydrogen Embrittlement Observed in Iron-Silicon Single Crystals" - A. S. Tetelman and W. D. Robertson, Transactions AIME, 224, 775, (1962)
5. "Martensitic Transformation in Single Crystals of Iron Alloys" - J. F. Breedis and W. D. Robertson, Acta Met., Accepted for publication, June 1962
6. "Direct Observation and Analysis of Crack Propagation in Iron-3 % Silicon Single Crystals" - A. S. Tetelman and W. D. Robertson, Acta Met., accepted for publication August, 1962
7. "Martensitic Transformation and Plastic Deformation in Iron Alloy Single Crystals" - J. F. Breedis and W. D. Robertson, submitted to Acta Met. August 1962
8. "An X-Ray Investigation of the Effects of Hydrogen in Iron" - A. S. Tetelman, C. N. J. Wagner, W. D. Robertson, Acta Met., 9, March 1961

In the attached Appendix the scope of each of the investigations is briefly outlined in the form of Abstracts.

Finally, another product of these investigations, in some respects more important than the results of research, is the training of competent investigators. Accordingly, it is interesting to have on record the present positions of the graduate students who have been engaged in these investigations.

David Dew-Hughes, D.Eng., Yale University, 1959, Demonstrator (Assistant Professor), Department of Metallurgy, Cambridge University, Cambridge, England.

John F. Breedis, D.Eng., Yale University, 1961. Post Doctoral Fellow, University of Illinois - 1960-61 and Research Associate Franklin Institute, Philadelphia.

Alan S. Tetelman, D.Eng. Yale University, 1961. National Science Foundation Post Doctoral Fellow, Faculté des Sciences, University of Paris 1961-62. Research Scientist, Ford Scientific Research Laboratory, Dearborn, Michigan.

Respectfully submitted,



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APPENDIX

THE MECHANISM OF HARDENING IN AGED ALUMINUM-COPPER ALLOYS

Single crystals were grown in aluminum-copper alloys containing from 3 to 5 per cent copper. These crystals were strained in tension after receiving heat treatments designed to develop CuAl₂ particles of varying sizes and distributions. Values of the critical resolved shear stress were found to be inversely proportional to the mean spacing between particles, in accordance with Orowan's theory. Fisher, Hart and Pry's theory, that dislocation rings formed around the particles during deformation make a large contribution to work hardening, has received confirmation from the experimental results on these crystals. While a crystallographically defined critical resolved shear stress was observed, deformation took place mostly by multiple slip, the external characteristics of the deformation being similar to those of polycrystalline material.

DISPERSED PARTICLE HARDENING OF ALUMINUM-COPPER ALLOY SINGLE CRYSTALS

Single crystals were grown in aluminum-copper alloys containing from 1 to 5 per cent copper. Crystals were tested in tension after an air-quench from the solution-treating temperatures. The aging behaviour of crystals containing 4 per cent copper was studied by X-rays and by hardness test.

Four per cent copper crystals were tested in tension after aging for various times at 130°C and 190°C. Crystals in all stages of aging obeyed the critical resolved shear stress law, though crystals containing Θ' particles deformed by multiple slip in the same manner as that previously found for over-aged crystals.

Strengthening due to the presence of Guinier-Preston zones was calculated by summing the contributions from Mott and Nabarro's coherency strain strengthening, Kelly's chemical strengthening and the strength of the residual solid solution. The calculated values of critical resolved shear

stress are in agreement with measured values on crystals aged for 2(GP-I) and 10(GP-II) days at 130°C and for 5(GP-I) days at 110°C.

A UNIFIED STRUCTURAL MECHANISM FOR INTERGRANULAR AND
TRANSGRANULAR CORROSION CRACKING

A unified, structural mechanism is proposed to explain the phenomena of intergranular and transgranular stress corrosion cracking. The dependence of failure on composition and stress in copper alloys and in stainless steels is analyzed in terms of two general conditions: (1), a chemically reactive path and (2), a mechanism of concentrating stress across the reactive path.

Intergranular fracture in a chemical environment is considered in terms of the normal stress distribution at the head of a group of dislocations piled up against a grain boundary. It is shown that the available data are consistent with the proposed model with respect to (1), a grain size dependence (2), stacking fault energy, which defines the number of coherent twin boundaries per grain and thereby the stress concentration at the boundary and (3), the existence of an endurance stress, approximately equal to the macroscopic yield stress, below which the stress concentration at the boundary is insufficient to cause fracture.

The nucleating sites for transgranular fracture in single crystals, and in polycrystalline aggregates that fail transgranularly, are shown to be Cottrell-Lomer barriers. A detailed crystallographic mechanism of fracture is proposed which depends on (1), orientation with respect to applied stress and (2), the strength of the barrier, which is related to the stacking fault energy. Available data are consistent with the model in predicting the composition dependence of transgramular fracture and, in favorable cases, the plane of fracture. The same mechanism is capable of explaining the dependence on composition and plastic strain of the transition from intergranular to trans-granular fracture, which is demonstrated with copper alloys.

THE MECHANISM OF HYDROGEN EMBRITTLEMENT OBSERVED
IN IRON-SILICON SINGLE CRYSTALS

The technique of decorating dislocations was employed to investigate plastic deformation and fracture resulting from precipitation of hydrogen in iron-3% silicon single crystals. It is shown that cracks are produced on {100} planes inside crystals quenched from a hydrogen atmosphere at elevated temperatures or cathodically charged with hydrogen at room temperature. Plastic deformation in the vicinity of cracks, observed as arrays of decorated dislocations, is in conformity with previously calculated stress distributions about a crack containing an internal pressure. The fracture characteristics of crystals containing internal cracks were evaluated at 25°C and -196°C and the results are related to the mechanism of hydrogen embrittlement.

MARTENSITIC TRANSFORMATION IN SINGLE CRYSTALS OF
IRON ALLOYS

The morphology and crystallography of the martensite transformation has been studied in single crystals of an Fe-16Cr-12 Ni alloy, representing an austenitic stainless steel. The transformation proceeds through a sequence of structures from f.c.c. \rightarrow h.c.p. \rightarrow b.c.c., originating with the h.c.p. structure in the {111}_A plane, which subsequently transforms to b.c.c. plates. A one surface analysis indicates that the habit plane of the plates is near {225}_A. Relative orientation of the three structures, lattice parameters, M_s temperature and reversion characteristics were determined and are considered with respect to the transformation mechanism.

DIRECT OBSERVATION AND ANALYSIS OF CRACK PROPAGATION
IN IRON - 3 % SILICON SINGLE CRYSTALS

Microcracks, 10^{-2} cm long on {100} planes, are produced by precipitation of hydrogen in iron-3 % silicon single crystals when hydrogen is introduced either by cathodic charging, or by quenching from a hydrogen atmosphere at a high temperature. Plastic deformation produced during growth of cracks can be observed as arrays of decorated dislocations in the volume of the crystals. The slip planes involved in plastic deformation around the tip of a crack have been identified and they are in accord with those predicted from theoretical considerations. Because of the discontinuous nature of the process of crack growth under internal pressure, it is possible to obtain values for the energy expended in plastic deformation, γ_p , as a function of crack length. For cracks about 10^{-2} cm long, γ_p is of the order of the intrinsic surface energy of the crystal, γ_s . Values for the distance to which plastic deformation extends from the tip of a stationary crack were measured and they compare favorably with values predicted from theoretical considerations.

MARTENSITIC TRANSFORMATION AND PLASTIC DEFORMATION
IN IRON ALLOY SINGLE CRYSTALS

The characteristics of plastic deformation of austenite single crystals have been studied in Fe-16Cr-25Ni, Fe-16Cr-12Ni, and Fe-30Ni alloys and related to the crystallographic mechanism of the martensite transformation.

The 16/25 alloy is completely stable down to 4°K and its plastic characteristics appear to be identical with other concentrated, f.c.c. solid solutions of low stacking fault energy. From 350°C to 200°C the plastic behavior of the 16/12 alloy crystals is similar to the stable 16/25 alloy.

Below 200°C transformation begins under stress and the critical resolved shear stress first increases and then decreases with temperature and appears to go to zero at -32°C due to the dilatation associated with the spontaneous transformation at this temperature. The transformation in the 16/12 alloy proceeds through the sequence of structures, f.c.c. → h.c.p. → b.c.c.c., originating on the {111}_A plane of maximum resolved shear stress. Other significant characteristics of the transformation were observed and related to the structure, namely: crystallographic reversibility of the transformation, stabilization by prior deformation, and isothermal transformation to h.c.p. and b.c.c products at room temperature following deformation at lower temperatures, and the effects of transformation products on the plastic properties of crystals.

Significant differences between the behavior of Fe-Cr-12Ni and Fe-30Ni crystals were observed and attributed to the difference in stacking fault energy and the associated differences in the transformation.

AN X-RAY INVESTIGATION OF THE EFFECTS OF HYDROGEN IN IRON

X-ray line broadening resulting from the cathodic charging of hydrogen into annealed and cold worked pure iron was investigated. Electrolytic charging at room temperature resulted in a broadening of all reflections, and Fourier analysis showed that the broadening was caused both by distortion and the presence of small, anisotropic domains. The Fourier coefficients, half widths, particle sizes, strains and recovery kinetics were found to be identical for iron that had been charged with hydrogen and for iron strained 5 per cent in tension; the apparent activation energy for recovery of line broadening, following either cathodic charging or a tensile strain of 5 per cent, is

42.0 \pm 5 kcal/mole in the temperature range 400-500°C. No significant change in lattice parameter was observed as a consequence of cathodic charging. All the evidence indicates that the effects of hydrogen on the X-ray diffraction characteristics of pure iron can best be interpreted in terms of plastic deformation, and there is no X-ray evidence to substantiate the recent proposal that hydrogen occupies tetrahedral interstitial positions in the iron lattice.